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UNIFORM VACUUM ULTRAVIOLET REFLECTING COATINGS ON LARGE SURFACES

by H. Herzig

Goddard Space Flight Center
Greenbelt, Md.

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ABSTRACT

A method for producing uniform vacuum-deposited films on large substrates from a single, small area evaporation source is described.

The procedure is being applied to the deposition of a uniform coating of magnesium fluoride on a 39-inch diameter vacuum-deposited aluminum mirror for use in the far ultraviolet. The magnesium fluoride restricts the oxidation of the aluminum and a uniformly high reflectance surface is obtained. Results of tests on a large number of two-inch square samples, arranged to duplicate the size and paraboloidal shape of the mirror, indicate that a reflectance of 82 ±2 percent at Lyman alpha can be expected.

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INTRODUCTION

Interest in the measurement of ultraviolet radiation of extraterrestrial origin has expanded in recent years as a result of the increasing ability to send instrument packages beyond the earth's ultraviolet-absorbing atmosphere.

Aluminum is the most important vacuum ultraviolet reflecting material known today, since it has the highest normal-incidence reflectance in the spectral region above 1000 A of any material studied. Its reflectance is approximately 90 percent or greater almost throughout the spectrum from the infrared down to 1400 A, falling to about 86 percent at 1026 A. In the region below 2000 A, however, the natural formation of aluminum oxide results in extensive degradation of reflectivity due to absorption. For example, at 1216 A the reflectance of aluminum will drop from 86 percent to 40 percent in a matter of hours when exposed to air, as the result of the formation of an oxide film only 20 A thick.

It is well known that this reduction in the reflectance of aluminum can be prevented by a protective coating of magnesium fluoride (Reference 1). With a magnesium fluoride overcoating 250 A thick, reflectances of 80 percent or greater can be achieved at 1216 A and the reflectance remains high at all longer wavelengths. This relatively small decrease below that of freshly evaporated aluminum is a result of absorption and interference by the overcoating. Below 1200 A, the absorption by the magnesium fluoride reduces reflectance sharply.

Aging of magnesium fluoride-on-aluminum coatings under normal storage conditions results in substantially no change in reflectance. A drop of 1-2 percent over a period of two years is typical (Reference 1).

Certain applications require uniform reflectance over large substrate areas. Since reflectance is highly sensitive to the thickness of the magnesium fluoride, in that increasing the thickness from 250 A to 380 A reduces the reflectance at Lyman alpha (1216 A) from 80 to 55 percent, it becomes necessary to control the deposit uniformity. Generally, thickness uniformity can be achieved by use of an evaporation source specifically designed to yield a uniform distribution or by altering the evaporant pattern from a source having a non-uniform distribution.

Some success has been achieved through the first approach using a ring source of tungsten wire loaded with the evaporant at even intervals (Reference 2). While powdered materials such as magnesium fluoride cannot readily be adapted to a ring source arrangement, it is probable that the same effect could be achieved with a small area source and a rotating substrate.

The technique used in the present work is based on the second approach, in which the thickness distribution from the magnesium fluoride source was altered by inserting a shutter between the source and the rotating substrate. The substrate requiring the vacuum ultraviolet reflecting coating was a 39-inch diameter paraboloidal mirror in the vacuum-optical-bench facility at GSFC. The mirror, which has a 7-inch diameter central hole, is used for checking optical experiments on satellites such as the Orbiting Astronomical Observatory (OAO). → 5

SHUTTER DESIGN

The technique of using a shutter to intercept varying amounts of a vapor stream was first utilized by Strong (Reference 3) to deposit a film of varying thickness on a spherical surface. The procedure was adapted by Behrndt (Reference 4) to the problem of producing vapor deposited films on numerous components simultaneously. While it is possible to rotate either the shutter or the substrate, the set-up of the evaporator used for this work made the latter alternative easier.

Although the mirror to be coated is paraboloidal, its radius of curvature at the vertex is sufficiently large, (154.4 inches) so that for the purpose of designing the shutter, its surface was considered flat.

The thickness of the coating on a flat substrate located above a point source or small area source is at a maximum directly above the source and decreases radially. The distribution can be calculated with the following equation from Holland (Reference 5)

$$\frac{t}{t_0} = \frac{1}{\left(1 + \frac{d^2}{h^2}\right)^2} ,$$

where t is the thickness at a distance d from the point directly above the source, t_0 is the maximum thickness (at $d = 0$), and h is the height of the substrate above the source.

A uniform film is obtained if, for all values of d less than d_{max} (at the edge), the amount of vapor passing the shutter is reduced to that at the edge. By using the following substitutions according to Behrndt (Reference 4),

$$\left(\frac{t}{t_0}\right)_{d_{max}} = y$$

and

$$\left(\frac{t}{t_0}\right)_d = x ,$$

the fraction p of the vapor stream which has to be intercepted at any point d less than d_{max} is given by

$$p = \frac{x - y}{x},$$

and the angle included by the shutter at that distance is 360p. Due to physical limitations, the shutter cannot be located in the same plane as the substrate; it must be installed at a height b less than h, i.e., closer to the source. The shape of the shutter is then calculated using the equation

$$d' = d\left(\frac{b}{h}\right),$$

where d' is the distance from the center on the shutter corresponding to the distance d on the substrate.

The shutter can be divided into any number of equal portions with the same results. In fact, the greater this number the smaller the magnitude of non-uniformity due to shadowing as a result of a non-integral number of rotations during the period of evaporation.

The actual shutter was composed of two sections as shown in Figure 1, because of ease of fabrication and the simplicity with which it could be supported within the vacuum chamber.

ALUMINUM COATING

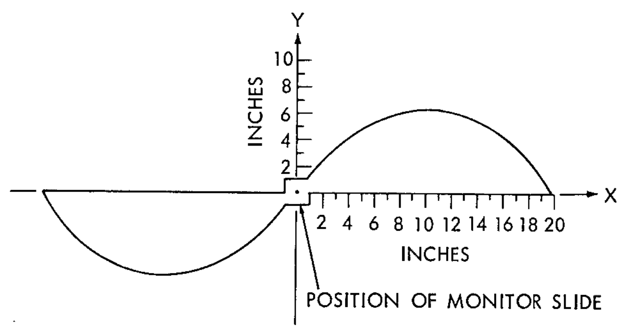

Figure 1 - Contour of shutter.

In order to obtain a nearly opaque coating of aluminum on a substrate of the stated size, it was necessary to use 16 tungsten filament sources loaded with aluminum staples. These were located in a circle concentric with the substrate with their longitudinal axes radially directed. It is not feasible to open the vacuum chamber between the aluminum and magnesium fluoride evaporations and still obtain a satisfactory reflective coating. Therefore, the shutter must be in position during the aluminum evaporation. Since the arrangement of filaments did not constitute a point or small surface source, it is not likely that the aluminum coating was uniform. However, satisfactory coatings can be produced with aluminum films ranging in thickness from 700 A to 900 A (Reference 1) and it was not necessary to include a mechanical device for moving the shutter out of the way during the aluminum evaporation. To prevent bare areas, the substrate was rotated at 90 rpm during the aluminum deposition as well as during the overcoating with magnesium fluoride.

MONITORING THICKNESS

The aluminum evaporation was monitored by measuring the change in reflectance of a 2-inch by 2-inch glass slide mounted on the underside of the shutter. A monochromatic light source with a wave length of 2537 A was used and the reflected radiation was picked up by a photomultiplier microphotometer. When the reflectance reached a maximum, the evaporation was stopped. Since the mirror has a 7-inch diameter hole in its center, the distortion of the shutter contour due to the presence of the monitoring slide does not affect the areas to be coated.

After the substrate had been coated with aluminum, the monitor readout was set to full scale and the magnesium fluoride evaporation begun. When the reading dropped to a value previously determined to correspond to the optimum thickness, the evaporation was halted.

RESULTS

Since it is not feasible to measure reflectance of a 39-inch diameter substrate, tests were run using a fixture containing a number of 2-inch by 2-inch glass slides arranged to duplicate the size and curvature of the mirror.

A preliminary test was made to determine whether or not the use of the shutter would result in the predicted thickness uniformity. Slides were partially masked and coated with magnesium fluoride. The masks were removed and the slides were coated with a flash of aluminum. The height of the magnesium fluoride step on each slide was measured with a high resolution interference microscope. A plot of thickness against radial distance is shown in Figure 2. Thickness is expressed as percentage of the fringe spacing, and is uniform over the entire substrate.

The next step was to measure the variation in reflectance over the substrate area when it had been coated with an ultraviolet reflecting coating, to determine whether some variable other than magnesium fluoride film thickness, such as angle of incidence, might affect the reflectance. These measurements were made with a one-meter normal incidence monochromator provided with a reflectance attachment. The results can be seen in Figure 3. The average reflectance was 82 percent with a variation of ± 2 percent over the entire surface.

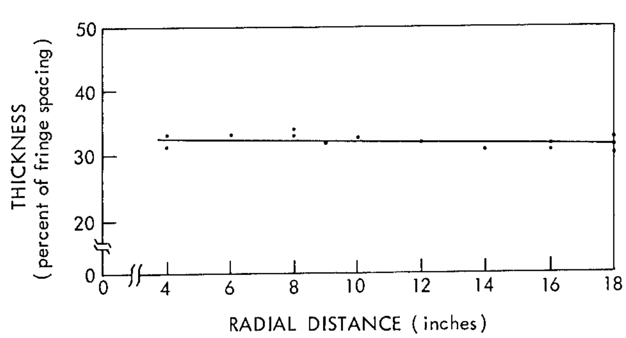

Figure 2 - Magnesium fluoride thickness distribution expressed as percentage of fringe spacing.

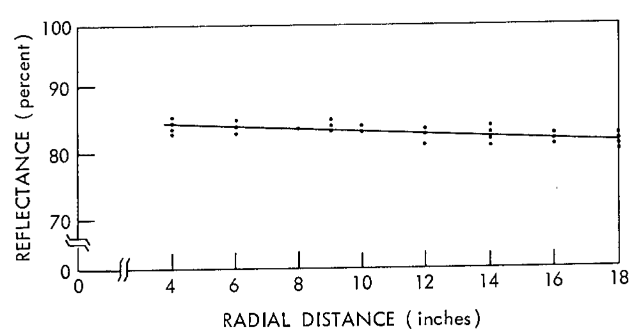

Figure 3 - Reflectance distribution at Lyman alpha.

The lines drawn through the data points in Figures 2 and 3 were determined by the method of least squares. It can be seen by examination that in each case the line slopes slightly upward toward the center of the substrate. It appears, therefore, that for the conditions of the tests, the reflectance uniformity is solely dependent on the uniformity of the magnesium fluoride deposit.

CONCLUSIONS

These results have shown that the technique of altering the pattern of flow of magnesium fluoride vapor from a small area evaporation source can be successfully applied to the problem of obtaining a uniformly reflective ultraviolet coating on large substrates. The small variations in reflectance can be attributed, in part, to the assumption of a flat substrate when designing the shutter. These can probably be decreased by using the more complex equation for thickness distribution on a spherical substrate (~~Reference 6~~), since this would more closely approximate the actual curvature of the paraboloidal mirror.

According to Behrndt (Reference 4), other factors which may contribute to non-uniform thickness are non-uniform heating of the melt, shadowing on the surface of the melt by the walls of its container, slag formation on the surface of the melt, and eccentricity of the source with respect to the axis of rotation.

Due to the geometry of the coating system used for these tests, the center of the source was displaced about 2 inches from the axis of rotation, which may have had some effect on uniformity. The effect of the other factors, if they existed, was probably minimized by rotation of the substrate rather than the shutter.

end

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REFERENCES

1. Madden, R. P., "Preparation and Measurement of Reflecting Coatings for the Vacuum Ultraviolet," in: *Physics of Thin Films - Advances in Research and Development,* ed. by G. Hass, New York: Academic Press, 1963, pp. 153, 172-3.

2. Bugenis, C., and Preuss, L. E., "Some New Experimental Data on Profile Families for Linear and Ring Sources," in: *National Vacuum Symposium of the American Vacuum Society, 10th, Boston, October 16-18, 1963, Transactions,* ed. by G. H. Bancroft, New York: Macmillan Co., 1963, pp. 374-378.

3. Strong, J., Maeher, H. V., Whitford, A. E., Cartwright, C. H., and Hayward, R., "Procedures in Experimental Physics," New York: Prentice Hall, Inc., 1938, p. 180.

4. Behrndt, K. H., "Films of Uniform Thickness Obtained From a Point Source," in: *National Vacuum Symposium of the American Vacuum Society, 9th, Los Angeles, October 31-November 2, 1962, Transactions,* ed. by G. H. Bancroft, New York: Macmillan Co., 1962, pp. 111-115.

5. Holland, L., "Vacuum Deposition of Thin Films," New York: John Wiley and Sons, Inc., 1956, p. 145.

6. Behrndt, K. H., "Thickness Uniformity of Rotating Substrates," in: *National Vacuum Symposium of the American Vacuum Society, 10th, Boston, October 16-18, 1963, Transactions,* ed. by G. H. Bancroft, New York: Macmillan Co., 1963, pp. 379-384.

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